REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

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1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE		3. DATES COVERED (From - To)		
20-02-2018	Final				
4. TITLE AND SUBTITLE		5a. C	ONTRACT NUMBER		
Test Operations Procedure (TOP)					
08-2-503 Low Volatility Agent Permeation	(LVAP) Swatch Testing	5b. G	5b. GRANT NUMBER		
		5c. P	ROGRAM ELEMENT NUMBER		
6. AUTHORS		5d. P	ROJECT NUMBER		
		5e. T.	ASK NUMBER		
		5f. W	ORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND	ADDRESS(ES)		8. PERFORMING ORGANIZATION		
U.S. Army Dugway Proving Ground			REPORT NUMBER		
West Desert Test Center			TOP 08-2-503		
(TEDT-DPW)					
Dugway, UT 84022-5000					
9. SPONSORING/MONITORING AGENCY NAME(, ,		10. SPONSOR/MONITOR'S ACRONYM(S)		
Policy and Standardization Division (CSTE	E-TM)				
U.S. Army Test and Evaluation Command			11. SPONSOR/MONITOR'S REPORT		
6617 Aberdeen Boulevard			NUMBER(S)		
Aberdeen Proving Ground, MD 21005-50	01		Same as item 8		
12. DISTRIBUTION/AVAILABILITY STATEMENT					
Distribution Statement A. Approved for pu	ublic release; distribution unlimited.				
42 CUDDI EMENTADY NOTES					

Defense Technical Information Center (DTIC), AD No.:

14. ABSTRACT

This Test Operations Procedure (TOP) provides the current standard methods for testing the permeation of low volatility chemicals, such as persistent nerve agent (VX), through swatches of materials. Swatches can be taken from clothing or equipment that is new, with or without pretreatment(s), and that was previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure(s). These procedures are designed to be used as part of an overall assessment program evaluating the material performance, manufacturing, and integration with other pieces of the protective ensemble.

15. SUBJECT TERMS

Low volatility agent permeation; LVAP; swatch; test operations procedure; TOP; chemical warfare agent; CWA; test cell; divinylbenzene; DVB; permeation; extraction efficiency; method quantification limit; MQL; persistent nerve agent; VX; quantifiable; liquid chemical agent permeation.

16. SECURITY CLASSIFICATION OF:			17. LIMITATION	18. NUMBER	19a. NAME OF RESPONSIBLE PERSON		
a. REPORT	b. ABSTRACT	c. THIS PAGE	OF ABSTRACT	OF PAGES			
Unclassified	Unclassified	Unclassified	SAR	39	19b. TELEPHONE NUMBER (Include area code)		



US ARMY TEST AND EVALUATION COMMAND TEST OPERATIONS PROCEDURE

*Test Operations Procedure 08-2-503 DTIC AD No: 20 February 2018

LOW VOLATILITY AGENT PERMEATION (LVAP) SWATCH TESTING

			<u>Page</u>
Paragraph	1.	SCOPE	2
<i>O</i> 1	1.1	Purpose.	2
	1.2	Objectives	
	1.3	Limitations.	
	2.	FACILITIES AND INSTRUMENTATION	4
	2.1	Facilities.	
	2.2	Equipment.	4
	2.3	Instrumentation	
	3.	REQUIRED TEST CONDITIONS	
	3.1	Test Planning.	
	3.2	Experimental Design.	
	3.3	Environmental Documentation.	
	3.4	Safety	9
	3.5	Quality Assurance (QA) and Quality Control (QC)	10
	4.	TEST PROCEDURES.	
	4.1	Test Method Outline	12
	4.2	Significance and Use.	12
	4.3	Calibration and Standardization.	
	4.4	Receipt Inspection.	13
	4.5	Agent Purity.	14
	4.6	Sampling/Analytical System	14
	4.7	Pretrial Procedures.	14
	4.8	LVAP Swatch Test Procedures	20
	4.9	Data Review.	23
	5.	DATA REQUIRED	24
	6.	PRESENTATION OF DATA	25
APPENDIX	A. C	GLOSSARY	A-1
	B. A	ABBREVIATIONS	B-1
	C. R	REFERENCES	C-1
	D. A	APPROVAL AUTHORITY	D-1

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1. SCOPE.

1.1 Purpose.

- a. This Test Operations Procedure (TOP) provides the current standard methods for testing the permeation of low volatility liquid chemicals, such as persistent nerve agent (VX), through swatches of air-permeable and air-impermeable material. These procedures may also be used with new methodologies for low volatility toxic industrial chemicals (TICs) and other emerging threats. These test procedures are performed under static conditions.
- b. The swatch material types may be single-layered or multilayered composites of inert, sorptive, or reactive layers. Swatches can be taken from clothing or equipment that is new, pretreated in the laboratory, or previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure, including simulated exposure. Swatches may also be taken from manufacturing sources, such as a bolt of material or a sample from a formulation that may be used for the manufacture of protective clothing or equipment.
- c. Use of the procedures in this TOP can provide relative ranking or screening information about the ability of candidate materials to resist liquid chemical agent permeation. The data may also support models that provide additional analyses; however, the creation or use of such models is beyond the scope of this TOP.
- d. Swatch testing is a required part of acquisition testing for the test and evaluation process. Swatch testing can also be employed as a quality control (QC) measure for production lot testing or surveillance testing of stored equipment.

1.2 Objectives.

- a. This TOP provides the basic information necessary to conduct and report swatch agent-resistance testing using low volatility liquid chemicals. This TOP also discusses the required facilities, equipment procedures, test and experimental parameters, and the data to be generated using these test methods. In cases where the Joint Capabilities Integration and Development System (JCIDS) requirements documents [the Capability Development Document (CDD) or the Capability Production Document (CPD)] are different from this TOP, the JCIDS document instructions will be followed.
- b. This document is a guide to preparing program-specific test plans or other test conduct planning documents (e.g., test plans, operations plans, letters of instruction, etc.).
- (1) Procedures described here may need to be tailored to address the particular objectives and requirements for a specific swatch test for specific low volatility chemicals.
- (2) Planning documents will include a rationale for any modification to the procedures in this TOP used for a swatch test.

1.3 Limitations.

- a. The procedures in this TOP are not sufficient to assess the ability of whole ensembles made from tested materials to protect the wearer. These procedures are designed to be used as part of an overall assessment program, evaluating the material performance, manufacturing, and integration with other pieces of the protective ensemble.
- b. These procedures cannot be used to determine permeation rates nor breakthrough times of the agent through the material.
- c. Results obtained by using these procedures may be used to statistically compare the agent resistance of materials tested during the same or a previous experiment. Historic swatch testing parameter information is included wherever applicable for reference and comparison with current and/or future threat test parameters. If comparison with previous data is planned, special caution must be taken to use the same test parameters to optimize the comparability of the data. In addition, a standard reference material must be used as one of the tested swatches.

 NOTE: During previous testing, including verification and validation (V&V) efforts, 10-mil-
- d. These procedures are not designed to yield results for correlation to specific medical or toxicological values or to protective system performance. Therefore, results obtained from following these procedures are not designed to be used to express an absolute protection value.
- e. The data obtained by these procedures are not designed to be correlated with specific field conditions.
- f. This method is solely a materials-level test that is applicable to testing swatches of air-permeable or air-impermeable materials under static conditions. This TOP does not apply to testing materials under stress-load conditions. This testing is limited to flat swatches of test materials. Testing materials with seams and zippers will require additional validation.
- g. These procedures measure the cumulative permeation during the test period as a single data point; as such it is not a near-real-time method.
- h. This TOP method may not be appropriate for testing contaminant-repellent materials because these materials do not absorb contaminants.
- i. This TOP may not be appropriate for chemical warfare agent (CWA) contaminants more volatile than VX, as vapor cross-contamination may occur around the edge of the swatch, leading to a false positive detection for permeation or background contamination. Steps should be taken to characterize this effect for each new contaminant.
- j. This TOP is limited to approved standards and procedures. Developments in practices, equipment, and analysis may necessitate establishing new testing baselines. Additionally, standards of performance must be adjusted as technologies advance. Test

thick latex has been used as a standard reference material. 1, 2, 3*

^{*}Superscript numbers correspond to those in Appendix C, References.

TOP 08-2-503 20 February 2018

procedures and parameters listed in this TOP require updating to accommodate new technologies in materials or in test instrumentation. Any updates should be described in the planning documents.

2. FACILITIES AND INSTRUMENTATION.

2.1 Facilities.

Facilities, instrumentation, and safety procedures used for swatch testing with chemical agents are strictly controlled. Additional discussion and requirements for facilities and instrumentation/equipment are included in the test procedures (Paragraph 4).

<u>Item</u>	Requirement
Chemical surety laboratory and chemical agent storage facility	Must be constructed to provide the capabilities needed for work with CWAs (only if CWAs are used during testing). Required capabilities include: (1) secure storage of agents, (2) general and specialized chemical analysis, (3) emergency response, and (4) hazardous waste storage and disposal.
Low volatility agent permeation (LVAP) swatch test fixture and control/data system	Must contain the test cells and provide the required test conditions. Must have the ability to control temperature from 10 °C above ambient to 50 °C (\pm 2 °C) and to record the test conditions every minute.
Preconditioning chamber	Must be verified to operate for 24 hr at specified conditions of 32.2±0.7 °C (90±2 °F) and 80±5 percent relative humidity (RH). The conditions will be measured and recorded using calibrated temperature and RH probes that are traceable to standards from the National Institute of Standards and Technology (NIST, Gaithersburg, Maryland).

2.2 Equipment.

	<u>Item</u>	<u>Requirement</u>
Test cell		The test cell consists of a polycarbonate Petri dish, a 47 mm polytetrafluoroethylene (PTFE swatch, a sorbent pad, a swatch, a 28-mm PTFE disk, and a cylindrical stainless steel weight, contained within an inverted 240-mL glass jar. A schematic is shown in Figure 1.

Requirement <u>Item</u> Cylinder weight Will consist of individually numbered 316 stainless steel cylinders, each with a mass of $454.0 \text{ g} (\pm 1 \text{ g})$ and a contact diameter of 28.7 mm. This mass and diameter will be capable of delivering 1 psi of pressure to a 6-cm² region. Should consist of a 3M[®] Empore^{®**} solid phase Sorbent material extraction disk or other comparable device (e.g., a latex contact sampler). In previous testing, a 47 mm divinylbenzene (DVB) extraction disk, part number SDB-XC, was used. 1, 2, 3 Latex swatch as standard reference 10 mil, medium-soft (40A durometer), natural material latex rolled sheets (part no. 85995K14; McMaster-Carr; Elmhurst, IL). The thickness tolerance is ± 0.002 in.

^{**} The use of brand names does not constitute endorsement by the Army or any other agency of the Federal Government, nor does it imply that it is best suited for its intended application.

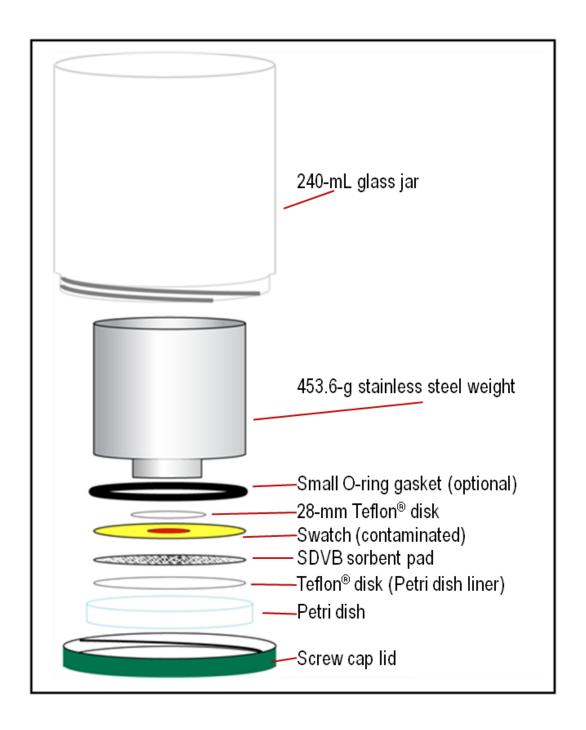


Figure 1. Schematic of the test cell.

2.3 Instrumentation.

<u>Parameter</u>	Measuring Device	Permissible Error of Measurement
Air temperature (normal target of 32.2 °C)	Thermocouple or other comparable device.	±0.7 °C
Relative humidity (RH) (up to 80 percent)	Humidity probe or other comparable device.	±5 percent
Contaminant application	Hamilton [®] Gastight [®] syringe (Hamilton [®] , Reno, Nevada) with a blunt tip needle or other comparable device capable of applying a liquid contaminant at a density of 10 g/m ² .	±10 percent of target
Chemical analysis of permeation samples (agent mass recovered in ng)	Gas chromatography (GC), Liquid chromatography- tandem mass spectrometry (LC- MSMS), or equivalent.	QC check samples and calibration standards should yield analytical results within 15 percent of the true value
Swatch thickness	Micrometer with a wide-foot design for fabrics and elastomers.	±0.0005 cm

3. REQUIRED TEST CONDITIONS.

- a. The swatch test fixture will contain the test cells needed for each trial and provide the required test conditions for each cell. The fixture data acquisition system will record the test conditions at least every minute.
 - b. Detailed descriptions of the test cell are found in Paragraph 2.2.

3.1 <u>Test Planning</u>.

- a. Test planning documents (e.g., test plan, etc.) must be developed for each test event to document the test objectives and criteria, experimental design (test matrix), test type and conditions, receipt inspection, swatch storage and tracking procedures, sample acceptance criteria, and data management plan.
- b. The capability documents [initial capability document (ICD), CDD, or CPD], the concept of operations (CONOPS), the system evaluation plan (SEP), and the test and evaluation master plan (TEMP) will be used to determine the data required, test objectives and criteria, and data analysis method to be used. If capability documents are not provided, the data required, test

objectives and criteria, and data analysis method will be coordinated directly with the customer and documented in the test plan.

- c. Test Objectives and Criteria. The test objectives and criteria will be used to determine the type of testing to be conducted as well as the test cell configuration, any conditioning and/or pretreatments of the swatch material, trial length, and extraction disk selection. To avoid confusion the term extraction disk will be used throughout this document, which is generally understood to be the DVB sorbent layer.
- d. The quantities of test materials and trials to be conducted will be determined based on the information collected from the capability documents, the SEP, and the TEMP, in coordination with the customer.
- e. A realistic sample size (number of trials) for each test material will be determined through review and coordination (based on test cost, data objectives, statistical power, value, and material availability) with the assigned operational test activity evaluator. In all cases, a design of experiment (DoE) method will be used to optimize test-material use and data output.
- f. Each test plan and other planning documents must be reviewed for technical accuracy and conformance with standing operating procedures (SOPs) applicable to the specific instrumentation used; to the item, system, or materials under test; and to the tests being conducted.

3.2 Experimental Design.

3.2.1 Trial Matrix.

- a. The trial matrix will be designed to balance the test materials among trials and fixtures as much as possible. The test material, positive and negative control swatch positions, and characterization swatches will be randomized within each trial. Characterization swatches serve as the baseline for the presence of contaminants.
- b. Randomization will be based on the swatch identification control number (SICN) assigned during the receipt inspection (Paragraph 4.4).

3.2.2 Sample Size.

The sample size (number of trials) will vary in accordance with (IAW) test program requirements. If not specified in a SEP, TEMP, or equivalent document, the sample size for each test/test condition will be determined based on the following factors:

- a. Test hypothesis.
- b. Selected statistical analysis technique.
- c. Confidence or significance level, including the alpha and beta levels.
- d. Expected data variability (based on previous test data).

e. A quantifiable difference that is meaningful, e.g., the amount of permeation needed to determine that one material has a better protective performance than another material.

3.2.3 Swatch Pretreatments.

Swatch pretreatments may be used to test how the agent resistance of a material will change when the material is exposed to various contaminants. The planning documents must state which pretreatments will be used, if any. TOP 08-2-501A⁴ Permeation Testing of Materials With Chemical Agents or Simulants (Swatch Testing) contains pretreatment procedures that should be reviewed. Common pretreatments are: petroleum, oil, and lubricants (POLs), water (fresh and simulated sea water), decontaminants, firefighting foam, insect repellant [N,N-diethyl-meta-toluamide (DEET)], body fluids (simulated sweat, urine, blood, and feces) and other compounds to which protective suits may be exposed during a mission. NOTE: If pretreated swatches are used during testing, it is strongly recommended that a pretest methodology investigation be conducted to determine whether the pretreatments affect the performance of the extraction disk of choice and to investigate possible interferences during sample analysis.

3.3 Environmental Documentation.

Testing will comply with all local, state, and federal regulations. Appropriate documentation will be prepared and submitted and approval will be received before testing begins.

3.4 Safety.

- a. LVAP swatch testing normally requires the handling and use of chemical agents. Such testing is strictly controlled by regulations [e.g., Army Regulation (AR) 385-10⁵, and Department of the Army (DA) Pamphlet (PAM) 385-61⁶]. Throughout testing, the primary emphasis will be on the safety of the fixture operators and other test personnel. Swatch testing with agent will be performed in an approved surety facility.
- b. For tests involving threat agents or simulants, qualified and trained operators and standard equipment will be used. The appropriate laboratory will be scheduled to conduct the test, and laboratory technicians will receive the appropriate training in system operations before testing begins.
- c. Applicable safety and surety regulations will be reviewed to ensure that all test procedures are in compliance.
- (1) Chemical agents are extremely toxic and exposure to them can be fatal; therefore, during testing with agents, the primary emphasis will be placed on safety.
- (2) Tests using chemical agents will be conducted IAW DA PAM 385-61⁶ and AR 50-6⁷, approved agent handling SOPs, and procedures specified in the planning documents.
- (3) Risk management and hazard analysis⁵ will be conducted and documented before the test plan is written.

(4) All test participants will thoroughly understand the test plan and applicable SOPs, and will acknowledge their understanding and training by signature.

3.5 Quality Assurance (QA) and Quality Control (QC).

3.5.1 QA Planning.

- a. A QA plan should be prepared for each test program to ensure that all controllable variables are controlled and that appropriate records are kept throughout the duration of testing. Variables that cannot be controlled must be identified in the test plan. Test variables include but are not limited to: purity and stability of the challenge agents or simulants, purity and stability of any decontaminants, calibration and maintenance of instrumentation and disseminators, accuracy and precision of the laboratory analysis, demonstration of operator proficiency, and quality and uniformity of all swatch samples.
- b. The condition of the test item (clothing or equipment) from which swatches are cut at the time of testing is an important test variable. The test items should be inspected IAW TOP 08-2-500⁸. Pretrial inspection of the swatches as a subtest is encouraged, but not required. Inspection data and certificates of compliance or similar documentation should be reviewed to ensure that exterior material surfaces, material finishes, and packaging, meet specifications. Generally, the materials should be tested in as-received condition, unless the condition does not meet the test specification. Swatch testing may be required periodically throughout the equipment lifecycle if normal wear or storage is shown to significantly affect protective performance.
- c. Test Conduct. Testing must always be conducted IAW approved test documentation, such as technical manuals, field manuals, equipment operating instructions, SOPs, the approved test planning directive, SEP, TEMP, and the test plan. Any deviations from the test documentation will be included in the test plan and/or report and approved by the appropriate authority.

3.5.2 OC Swatches.

- a. Control swatches demonstrate control of all parameters affecting test results across trials and verify that the analysis was not affected by the presence of positive and negative interferents. Each trial will have two sets of control swatches, positive and negative.
- b. At least one positive control swatch will be included for each trial. The purpose of the positive control will be to demonstrate that the entire test process was working properly. This will be accomplished by using a swatch of a standard reference material with a known measurable permeation mass. During previous testing, a 10-mil thick latex rubber has been successfully used as the positive control swatch material^{1,2,3}. The positive control swatch will be contaminated with the challenge chemical at the level specified in the detailed test plan.
- c. At least one negative control swatch from each material will be included for each test day. The purpose of the negative control will be to demonstrate that the entire process was working properly, and that any cross contamination would not register as a permeation. Such

cross contamination can occur from test cells or tools. The negative control swatch will be randomly chosen from among the test materials each day and will be processed the same as the test swatches except it will not be contaminated with the challenge. A measured permeation concentration would indicate an issue with the test process and will require a process review.

3.5.3 Agent Challenge QC.

The agent challenge will be verified by spiking a PTFE disk with the target contamination density and extracting the disk with high performance liquid chromatography (HPLC) grade acetone to recover and measure the agent (VX used in validation of these procedures) dispensed. Other agents may require other extraction solvents. Data, such as solvent volume and extraction time, will be collected during the analysis procedures. The analysis will ensure that the agent dissemination instrument, the solvent volume control, and the analytical capabilities are operating properly. At least two PTFE disks will be used for each test, one at the beginning of the operation, and a second at the end. During large test processes, a third PTFE disk may be included in the middle of the sample queue.

3.5.4 Data Management Plan.

a. The data management plan will ensure that all data collected will be reviewed and will receive a QC check by qualified individuals. This plan will provide for the timely and accurate processing of test data for submittal to the customer.

b. Data Control.

- (1) All data will be handled using chain of custody (CoC) procedures. An example of CoC procedures is found in Paragraph 4.4.
- (2) All test, analytical, and environmental QC data will be audited to ensure the results are within the parameters detailed in the organization's SOPs and approved planning documents.

c. QC Review.

- (1) All test and data management procedures and QC data should be verified throughout testing as specified in Paragraph 3.5 of this TOP, or test planning documents.
 - (2) QC data will be recorded and reported as required by the specific test program.
- (3) All aspects of testing will be performed with an emphasis on acquiring quality results that are credible and verifiable.

4. TEST PROCEDURES.

4.1 Test Method Outline.

- a. Receipt inspection will be conducted on the test items or swatches, the preconditioning jar and test cell, and any other materials to document the as-tested material conditions (Paragraph 4.4).
- b. Agent purity will be analyzed and recorded (Paragraph 4.5), and the sampling and analytical systems will be calibrated (Paragraph 4.6).
- c. The extraction and uptake efficiency of the extraction disk will be determined (Paragraph 4.7.1)
 - d. Swatches will be cut and pretreated, as required (Paragraph 4.7.2).
- e. The swatch and the test cell will be environmentally preconditioned [i.e., conditioned with the correct test temperature and RH] within the fixture, if required (Paragraph 4.7.3).
- f. Agent challenges will be applied to the swatches. The test cells will be assembled and sealed with the swatches inside (Paragraph 4.8.1) and the swatch trial will begin.
- g. The trial will end and the procedures will be followed to collect and analyze all remaining sampling system data (Paragraph 4.8.2).
- h. Test data will be reviewed for consistency and acceptability during trial execution or as soon as possible after testing (Paragraph 4.9).

4.2 Significance and Use.

Application of the procedures in this TOP can provide relative ranking or screening information about the ability of candidate materials to resist chemical agent permeation. The data may also support models that provide additional analyses; however, the creation and use of such models is beyond the scope of this TOP.

4.3 Calibration and Standardization.

- a. Most chemical analytical equipment (e.g., GCs, LCs, etc.) will be calibrated by following the general chemical analytical calibration guidelines that are specified in the approved SOPs of the testing installation.
- b. Standards and QC samples will be prepared using the procedures specified in the approved SOPs or practices of the testing installation.

4.4 Receipt Inspection.

- a. All test items will undergo a receipt inspection as part of the QA/QC process (Paragraph 3.5.1.b). Test item CoC procedures must be implemented immediately upon receipt. The receipt inspection procedures outlined in the TOP 08-2-500⁷ will be followed.
- b. Both sides of each test item will be inspected for rips, tears, stains, and other damage using visual inspection or light transmission methods. Any abnormal characteristics of the test item will be noted, the observations recorded in a receipt logbook, and the customer will be consulted on any path forward for that item. Surfaces will be inspected for foreign materials not normally present on the item (dust, mud, or markings). Foreign materials may be removed, as much as possible, by brushing or vacuuming. The removal of foreign materials will minimize the bias that could cause analytical instruments to overestimate or underestimate the true agent permeability of the material being tested. Where feasible, a photographic record (with metric scale) will be made of all damaged swatches.
- c. As part of the receipt inspection and test item tracking process, each test item will be given a test item control number (TICN). Each swatch cut from the test item will be given a SICN based on the TICN. These numbers will be used to uniquely identify each test item by test program and to track test items and swatches throughout the test process.
- d. The unique SICN will identify each swatch by the location on the original test item from which the swatch was cut, as well as by the test program, material type, challenge agent, and trial sequence number. The CoC form for each swatch will be initiated after the swatch is cut from the test item.
- e. Swatches and test items will be stored until needed for trials in a clean, controlled environment. Limiting access to the storage location is particularly important for materials that are sorptive or reactive.
- f. Once individual swatches are sorted for testing, each will be placed in a sealable plastic bag that has been labeled with the SICN.
- g. Swatches comprising multilayered or composite materials will be kept with the layers together and in the order and orientation that would occur in the material as it would be worn. A small, clear plastic bag is recommended for storing multilayered or composite-material swatches.
- h. The CoC document for each item/swatch must be updated at the time custody is transferred for further processing. The CoC document should contain:
 - (1) The TICN/SICN for the item.
 - (2) The date and time the item/swatch was received.
 - (3) The signature of the individual relinquishing custody.
 - (4) The signature of the individual receiving custody.

- (5) A brief description of the operation conducted with the item/swatch.
- (6) The current location of the item/swatch.
- i. The preconditioning and test jars will also be receipt inspected. A TICN will be assigned to each jar when it is used to hold a swatch.
- j. In some cases, precut swatches may be received instead of clothing items or panels of material. If precut swatches are received they will be inspected IAW Paragraph 4.4.b, given a SICN, and tracked with CoC procedures.

4.5 Agent Purity.

The purity of the chemical agents used will be recorded as test data. Single agents used for swatch testing will be at least 90 percent pure. If a weapons-grade agent mixture is specified for the test, the purity of the defining agent will be measured and recorded as test data. The agent purity will be measured IAW the test facility's SOP.

4.6 <u>Sampling/Analytical System.</u>

Setup and preparation will depend on the sampling/analytical system used for each specific test and is subject to local SOPs. However, it should be noted that temperature and humidity probes must be calibrated before use. In addition, a calibration or QC check should be included at the beginning and end of the analytical sample queue to verify that the sampling/analytical system remains in calibration for the test sample analysis.

4.7 Pretrial Procedures.

4.7.1 Extraction and Uptake Efficiencies.

a. It is required that the efficiency performance of the extraction disk be characterized by efficiency testing with the contaminant of interest. This characterization needs to be established once for each combination of contaminant, sampler, contact time, and concentration range. Historically, three control samples and five test samples have been used for efficiency testing. Each lot of extraction disks must have a QC check performed to determine if extraction efficiency has changed since the last purchase.

b. Extraction Efficiency.

(1) During extraction efficiency testing, the extraction disk will be handled IAW the test requirements. Disks could be prepared IAW the manufacturer instruction for "wet" preparation, or they could be used as packaged. Each disk will be placed in the bottom of a test cell and spiked with a standard solution of the contaminant in an appropriate solvent. The volume of the solution will be predetermined IAW the requirements documentation. After spiking, 20 mL of solvent will be added, the jar will be closed, and the disk will be extracted for at least 30 min. An aliquot of the extraction solution will be removed for analysis.

- (2) The dissemination mass will be measured by spiking the same volume of the prepared solution into 20 mL of extraction solvent.
- (3) The average mass disseminated or extracted will be calculated using Equation 1. Using the data from the average mass disseminated (from control samples) and the average mass extracted (from test samples), the extraction efficiency is calculated using Equation 2.

$$\overline{m} = \frac{1}{N} \sum cV$$
 Equation 1

Where

 \overline{m} = the average mass disseminated or extracted

N = number of spike disks

c = the measured concentration

V = the volume of solvent used in the extraction or in the disseminated solution

$$e_e = \frac{\overline{m_e}}{\overline{m_s}} * 100 Equation 2$$

Where

e_e = the extraction efficiency

 $\overline{m_e}$ = the average mass extracted calculated using Equation 1

 $\overline{m_s}$ = the average mass disseminated calculated using Equation 1

Uptake Efficiency.

- (1) During uptake efficiency testing, a PTFE disk will be spiked with a solution of the contaminant in solvent. Once the solution has evaporated, the PTFE disk will be covered with the prepared extraction disk and the 454.9-g weight. Another PTFE disk or an aluminum foil disk will be used as a spacer between the extraction disk and the weight. The spiked PTFE and extraction disks beneath the weight will be sealed in a jar and allowed to incubate at the environmental condition and for the amount of time specified in the requirements document.
- (2) Once the required contact time has passed, the extraction disk and the spacer will be extracted together with an appropriate solvent. The originally spiked PTFE disk will be extracted in a separate jar containing solvent.
- (3) The dissemination mass will be measured by spiking a PTFE disk with the same volume of the prepared solution, allowing the solvent to evaporate, followed by extraction in 20 mL of solvent.

- (4) Uptake efficiency will be calculated using Equations 1 and 2. The extraction of the disk provides the extraction mass, and the PTFE control spike serves as the disseminated mass.
- (5) It should be noted that the uptake efficiency measurement is affected by the extraction efficiency, i.e., the sorbent pad could be very efficient at uptake, but if the subsequent extraction efficiency is poor, the overall uptake efficiency measured will also be poor. The extraction of the original PTFE disk spiked and then contact sampled with the extraction disk, provides information on the residual contaminant present that was not sorbed by the extraction disk.
- d. The individual sample result, including analyzed mass and efficiency results for extraction and uptake will be reported in the test documentation.

4.7.2 Swatch Preparation.

- a. Swatch Cutting. The swatch material will be cut into swatches with a 5-cm diameter using a hydraulic die cutter or similar equipment. Swatch thickness should generally be less than approximately 0.32 cm; however, there is some flexibility for greater thicknesses.
- (1) Each swatch will be given a SICN and placed in a sealed plastic bag until needed for testing. The bag will be marked with the SICN. The SICNs will be recorded.
- (2) The swatches will be inspected and measured. The thickness of each swatch will be measured using a micrometer designed for use with fabrics and elastomers and the measurement will be recorded. Measurements will be taken at the center of each swatch and at two points diametrically opposite from each other on the swatch edges. The three measurements will be averaged and recorded.
- b. Swatch Pretreatment. If swatch pretreatments are used, the pretreatment process will be conducted before the swatches are assembled into the test cells. **NOTE**: If swatches are pretreated, then the procedures in Paragraph 4.7.3 will not be conducted.

4.7.3 Swatch Preconditioning.

- a. The preconditioning chamber will be set up as follows:
- (1) The chamber will be run for at least 24 hours, with temperature and RH readings captured electronically at 1-minute intervals to confirm chamber environmental control can be maintained at the temperature and RH specified by the requirements document for the 24-hour swatch preconditioning.
- (2) The temperature and RH will be measured and recorded. <u>NOTE</u>: Exposure to high RH can result in excess water sorption on sorbtive swatch material that will not readily desorb during the preconditioning period.

(3) A typical preconditioning chamber will be set up with swatch holders as shown in Figure 2. An individual swatch holder is shown in Figure 3. An alternate preconditioning chamber setup (using clamps instead of jars to hold the swatches) is shown in Figure 4.

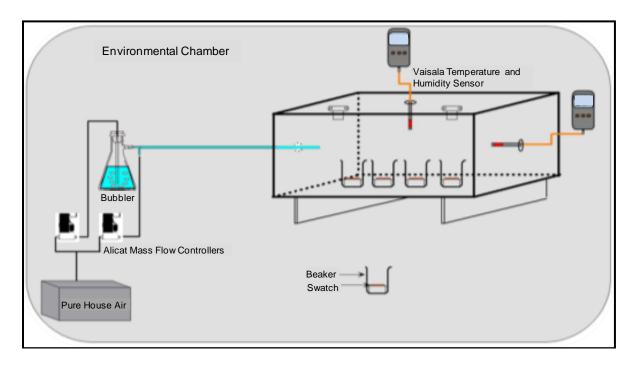


Figure 2. Swatch preconditioning chamber.



Figure 3. Swatch preconditioning jar, mesh platform, and sample swatch.



Figure 4. Alternate swatch preconditioning chamber, with clamps tack-welded to a screen.

- (4) A humidified airstream will be generated. During previous testing¹, a large bubbler was filled with deionized (DI) water and used to generate humidity. Other methods of generating humidity, such as NafionTM tube systems, are also acceptable.
- (5) A mixture of dry and humidified air will be delivered to the sealed swatch preconditioning chamber through the use of mass flow controllers.
- (6) Two temperature and humidity probes will be used to monitor temperature and humidity in the sealed chamber; this will also be used to record the temperature and RH over the entire preconditioning period.
- b. If using individual swatch holders (preconditioning jars), as shown in Figure 3, the swatches will be weighed and placed inside 240-mL jars (with the lids removed) before being placed in the preconditioning chamber. The jars minimize handling of the swatches and can be capped before being removed from the chamber to help preserve preconditioning. If using the

alternate chamber design (Figure 4), the swatches will be placed within the clamps. The clamp position number will be recorded as part of the data quality system.

- (1) Each jar will contain a platform made from a mesh screen on which the swatch will be placed to maximize exposure of both sides to the chamber temperature and RH.
- (2) The jars containing a swatch will be placed in the preconditioning chamber (uncapped) at the required temperature and RH for a minimum of 24 hours before agent application. The preconditioning start time will begin when the target temperature and RH have been achieved.
- (3) At the conclusion of preconditioning, the preconditioning chamber will be opened and the lids will be immediately screwed onto the jars to protect the swatches.
- c. After preconditioning, it is suggested that the swatches be quickly removed from the jar, weighed, and returned to the jar, to determine the amount of moisture sorption during preconditioning. Personnel should minimize the amount of time the swatch is outside the jar.
 - **NOTES**: 1. Weighing the swatches before and after preconditioning is required as a verification step. This characterization will be necessary for each swatch type. Once the total water uptake of a test material is well characterized, the weighing may not be necessary. If weighing the preconditioned swatches is required for a test, then it is recommended that a statistical sampling of at least five swatches be weighed.
 - 2. Previous characterization testing showed that swatches started to lose weight quickly upon removal from the preconditioning chamber. The length of time the swatches are outside the preconditioning environment should be kept to a minimum to reduce moisture loss before agent application.
- d. The swatches will not be removed from the jars until immediately before agent application.
- e. In very dry environments, it may be necessary to keep sorbent swatches within the humidity-controlled space until that particular swatch is ready to be contaminated for testing. The humidity protection procedure should be documented as part of a verification procedure.

4.7.4 Test Chamber Preparation.

- a. The LVAP swatch test fixture should be composed of a temperature-controlled test chamber. The temperature in the fixture should be characterized before testing to demonstrate uniformity across the chamber.
- b. During permeation testing, at least one thermocouple located inside the test chamber will be used to measure and record the temperature at intervals of at least 1 minute. RH is not controlled in the test chamber because the swatches are in sealed jars.

c. The test cells, the other components used in testing, and cylinder weights will be placed in the test chamber for a minimum of 2 hours before the test to allow the cells and weights to reach the test temperature. The weights will be removed from the test chamber only to place them on the swatches in the test cells, which will then immediately be placed back inside the test chamber.

4.8 LVAP Swatch Test Procedures.

4.8.1 Agent Application and Test Cell Assembly.

- a. For the LVAP test, Petri dishes will be preloaded with 47-mm PTFE disks (underneath the extraction disk) and 47-mm extraction disks. If using wetted extraction disks, the disks should remain in a sealed jar and be placed onto the Petri dish just before each individual swatch is added. Air-impermeable swatches may also be preloaded into the Petri dish. Air-permeable swatches will remain within a closed jar until just before the time of contamination.
- b. Previous testing^{1, 2} has shown the presence of contaminants in the DVB extraction disk after a long period of time (24 hours) and elevated temperature trials. This may be addressed with the use of the O-ring gasket, as shown in Figure 1. The O-ring specifications are: Buna-N O-ring, part no. 224N70; Paramount Packing and Rubber; Baltimore, MD; with a nominal outer diameter of 2.0 in. and a nominal inner diameter of 1.75 in. The O-ring serves as a gasket, sealing against the stainless steel weight to prevent vapor cross-contamination.
- c. To contaminate a particular swatch, a single jar containing the contact weight will be removed from the LVAP swatch test fixture. The jar will be opened, the weight removed, and the jar lid inverted. The Petri dish containing the PTFE disk will be placed on the inverted lid. The dry extraction disk will be placed on the PTFE disk, followed by the swatch, using a pair of disposable forceps. If the swatch is highly textured, contains a seam, or contains some other material that may promote liquid wicking over the edge, an annular ring of detector paper may be used between the extraction disk and the swatch as a visual indicator of a wicking event. A 2-in. O-ring gasket will be placed within the Petri dish. The swatch will be contaminated with six 1-µL drops of VX, as shown in Figure 5. This entire process is demonstrated in Figure 6.

 NOTE: During previous testing¹ the agent was applied using a Hamilton® Gastight® syringe, a 50-µL cemented needle (product 80975), a 22-gauge blunt-tipped needle, Type 3, and a repeating Dispenser (product PB-600), all manufactured by Hamilton® company, Reno, Nevada).

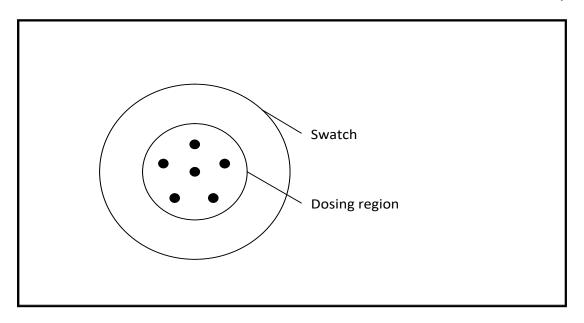
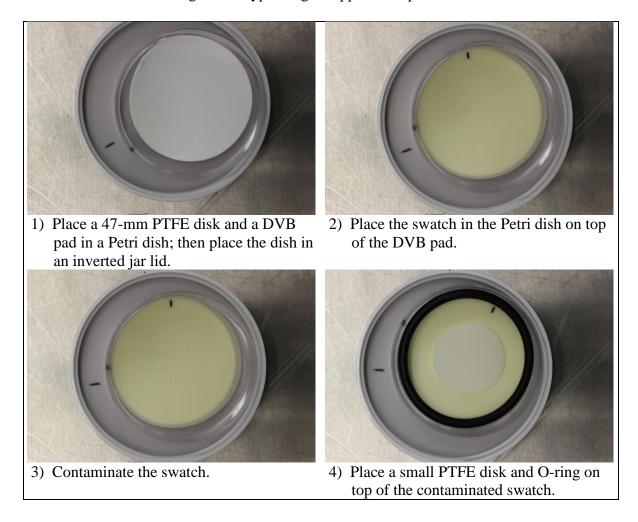
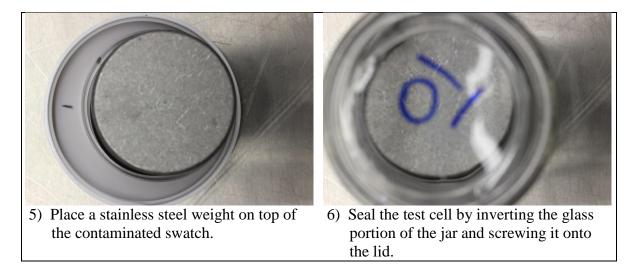


Figure 5. Typical agent application pattern.





NOTE: PTFE – polytetrafluoroethylene; DVB – divinylbenzene.

Figure 6. Step-by-step procedures for assembling a low volatility agent permeation (LVAP) swatch test cell.

- d. The contaminated swatch will be photographed. The contaminated area will be covered with a 28-mm PTFE disk placed with a second set of disposable forceps. The stainless steel weight will be applied and the glass portion of the jar will be inverted and screwed into the jar lid to seal the test cell. The whole test cell will be placed into the LVAP swatch test fixture. The contamination time will be noted on the run sheet. This process will be repeated every 2 minutes (recommended time) using a timer to mark the appropriate spiking time.
- e. After all swatches are contaminated, the fume hood will be secured IAW necessary safety, security, and surety requirements.

4.8.2 <u>Sampling and Analysis</u>.

- a. After 24 hours of contact, each test cell will be removed from the LVAP swatch test fixture for individual processing. The cell will be disassembled. The stainless steel weight will be placed on a funnel over a waste container and rinsed with solvent to remove any contamination from the contact region.
 - b. The swatch will be photographed again to document the extent of agent spread.
- c. Using disposable forceps, the layers within the cell will be separated. A new set of forceps will be used to pick up the extraction disk and place it into a jar preloaded with solvent for extraction. Record the time that the extraction disk is removed from the cell and placed in the extraction solvent. New forceps will be used for each swatch cell. **NOTE:** It is extremely important that the forceps be used as close to the edge of each layer as possible to avoid removing contamination and adding variability to the analytical results. The glass jar, lid, Petri dish, and PTFE disk pieces will be disposed of as hazardous waste IAW approved SOPs.

- d. Once the solvent has evaporated from the stainless steel weight, the weight will be placed into a new jar, and the jar will be inserted into the LVAP swatch test fixture in preparation for the next test.
- e. At the end of the disk extraction time (recommend at least 10 minutes), the extraction jar will be swirled gently for 15 seconds. After extraction, two aliquots of extract are transferred to pre-labeled autosampler vials. One aliquot will be used for immediate analysis and the other archived for future analysis if needed. All extracts should be stored at \leq 4 °C and analyzed within 14 days.
- f. The analyst assigned to test the samples will review the CoC accompanying the samples to ensure accuracy of the codes and completeness of the sample set. After documenting that the analytical samples have been properly labeled, the analyst will log each sample into the laboratory database by sample code, date and time received, storage location, and name of the sample custodian relinquishing the samples for analysis. The analyst will then sign the CoC form for custody of the samples. **NOTE**: The COC form for each test sample generated during the test will be made part of the data package.
- g. The samples will then be quantitatively analyzed for agent concentration using a gas chromatography (GC)/flame photometric detector (FPD) or LC-MSMS or other appropriate method.
- h. LVAP Test Fixture Monitoring. The environmental parameters of the test chamber will be monitored to ensure they stay within the specified tolerance ranges for the duration of the trial. Environmental parameters will be recorded at least every minute.

4.9 Data Review.

The test officer (TO) will review all data obtained for consistency and acceptability during trial execution or as soon as possible after the test. Specifically, the following items will be reviewed:

- a. The agent challenge QC check will be reviewed to ensure the target challenge concentration was met, if applicable.
- b. The analytical results for positive control swatches will be reviewed to determine if the swatches remained within control limits.
- c. The negative control swatches results data will be reviewed to determine if cross contamination occurred in their test cells.
- d. Analytical QC and calibration data will be reviewed to determine if the values are within limits specified in the planning documents and SOPs.
- e. The chamber temperature and humidity data will be reviewed to ensure they stayed within the accepted control limits during the trial and conditioning periods.
 - f. Review all photographs.

- g. Review the swatch weight data for any anomalies.
- h. Review pre-conditioning data for anomalies.

5. DATA REQUIRED.

The following data are required for each trial.

- a. TICN for each panel of material or clothing item received (unless swatches are received directly), and the location the swatch is taken from.
 - b. SICN of each test swatch.
 - c. Test item and swatch inspection data by TICN/SICN.
 - d. Swatch weights by SICN.
 - e. Trial identification.
 - f. Conditioning chamber used, test chamber identification, and laboratory used.
 - g. Swatch pretreatments, if any.
 - h. Swatch agent challenge (drop volume and pattern).
 - i. Agent application QC validation.
 - j. Results of the negative and positive controls.
 - k. Mass of agent (µg) collected.
 - 1. Calibration and QC sample results from all chemical analyses.
 - m. Agent purity analysis results.
 - n. Trial duration.
- o. Temperature and RH for all environmental control fixtures during preconditioning and trial execution. These values will be recorded.
 - p. Photographs.
- q. Operator-annotated comments for any observed anomalies during execution of each trial, including observations of each swatch recorded by SICN.

6. PRESENTATION OF DATA.

a. In the test report, a summary data table is required to show the average permeation for each material type, along with the standard deviation and relative standard deviation (RSD). The average may be the mean or geometric mean as appropriate, based on the normality of the data. A comprehensive table will include the test number, analytical measured concentration (relating to the permeated mass), analytical dilution factor, and total permeated mass for each swatch. Examples of the data tables are shown in Tables 1 and 2. A plot showing the individual data points will be provided as well, as shown in Figure 7.

TABLE 1. SUMMARY DATA TABLE EXAMPLE

Material	Gasket Present?	n ^a	Geometric Mean (ng)	StDev ^b (ng)	RSD ^c (%)	Measured Breakthrough (%) ^d
Butyl	Yes	7	BQL ^e	N/A ^f	N/A	N/A
	No	6	440.4	660	149.9	0.007
Latex	Yes	10	4.57×10^{6}	181	4.0	75.6
Latex	No	10	4.60×10^{6}	116	2.5	76.0
Neoprene	Yes	10	9.67×10^{5}	56	5.8	16.0
	No	10	9.81×10^{5}	69	7.1	16.2

^aNumber of replicates.

^bStandard deviation.

^cRelative standard deviation.

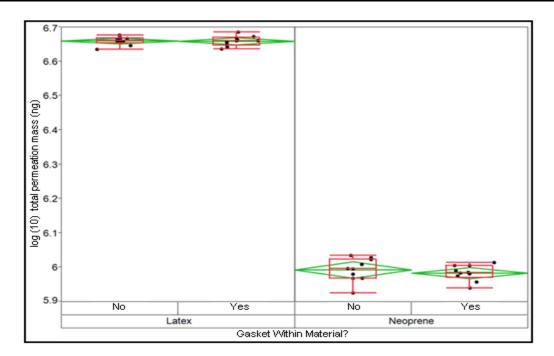
^dBreakthrough amount compared to the agent QC challenge.

^eBelow quantifiable limit.

^fNot applicable.

TABLE 2. COMPREHENSIVE DATA RESULT TABLE EXAMPLE

			Cell	Analytical		Mass of
Swatch		Trial	Position	Concentration	Analysis	Permeation
Material	Gasket?	Identification	Number	(ng/mL)	Dilution Factor	(ng)
			2	2.34×10^{5}	1000	4.68×10^{6}
			4	2.35×10^{5}	1000	4.70×10^{6}
		D	8	2.30×10^{5}	1000	4.60×10^{6}
			11	2.19×10^{5}	1000	4.38×10^{6}
	Ma		13	2.30×10^{5}	1000	4.59×10^{6}
No	NO	K	4	2.32×10^{5}	1000	4.64×10^{6}
			8	2.22×10^{5}	1000	4.43×10^{6}
Latex	Latex		21	2.28×10^{5}	1000	4.56×10^{6}
		32	2.33×10^{5}	1000	4.65×10^{6}	
			38	2.38×10^{5}	1000	4.76×10^{6}
			3	2.37×10^{5}	1000	4.75×10^{6}
	Yes	D	23	2.25×10^{5}	1000	4.50×10^{6}
			24	2.13×10^{5}	1000	4.25×10^{6}
			34	2.16×10^{5}	1000	4.32×10^{6}
			39	2.30×10^{5}	1000	4.61×10^{6}



NOTE: Each black dot indicates an individual swatch data point. The red lines indicate the standard quantiles as box plots. The green lines indicate the mean, the analysis of variance (ANOVA) overlap, and the 95 percent confidence interval bounds.

Figure 7. Swatch data plot example.

- b. The tables will include the pretreatment (if applicable), the test date, the SICN, and the average temperature and humidity.
- c. Each data set will be examined for outliers, but all data will be reported together with the rationale for eliminating any points from subsequent statistical analyses.
- d. The test plan must include information on how to handle sampling periods with results below the experimentally derived method quantification limit (MQL) for the individual laboratory. The common practice in acquisition testing has been to set below-MQL values to half the MQL because models and statistical processes cannot handle zero values and a below-MQL determination does not mean no contaminant is present. NOTE: There cannot be a predetermined or preset program or interlaboratory MQL imposed on a laboratory by requirements documents. MQLs are determined at the laboratory level.
- e. Because MQLs are determined at the laboratory level, issues may arise when data from different laboratories are compared. To resolve this issue, it is recommended that a program quantification limit (PQL) be set at the level of the highest performing laboratory MQL. This value would be used as the MQL at each individual laboratory (Paragraph 6.d).
- f. Procedures for statistical analysis of the data will be developed as specified in the planning documents. Analysis procedures must include:
 - (1) Method of addressing the program criteria.
 - (2) Type of analysis.
 - (3) Procedures for analysis of data points below detection limits.
- (4) Specifications of statistical values to be used (e.g., confidence, probability, power, etc.).
 - g. Permeation Calculations
- (1) The agent permeation will be calculated based on the analytical result ($\mu g/mL$), the extraction solvent volume (20 mL), and the swatch surface area (6 cm²).
 - (a) Flux of agent permeating can be calculated using Equation 3.

$$J = \frac{cV}{A}$$
 Equation 3

Where,

J =the mass flux (µg/cm²)

c = the concentration of the extracted solution as measured by analytical equipment

V = the extraction solvent volume (mL)

A =swatch surface area (cm 2)

TOP 08-2-503 20 February 2018

(b) The geometric mean values for permeation flux, standard deviation, RSD, and percent mass breakthrough compared with initial contamination will be calculated for all test materials.

APPENDIX A. GLOSSARY.

Breakthrough A mass or concentration of a challenge contaminant found on the

initially clean side of a contaminated test swatch, indicating that the contaminant has permeated or penetrated the material being tested. Breakthrough threshold levels are defined for each test program and should be specified as needed in the test plan or other test planning

documents.

Candidate material A nonstandard material, supplied as a flat piece, a bolt, or a

manufactured item, from which swatches may be cut for testing.

Cell A test cup used to hold a test swatch for permeation or penetration

testing. Cups have top and bottom sections with the test swatches

attached to the lower section.

Challenge The chemical agent or agent simulant (or other chemical compound)

that is placed on one side of a test swatch. The challenge can be in liquid or vapor form. The challenge amount is usually stated in planning documents in units of g/m^2 for liquid or mg/m^3 for vapor.

Permeation The process by which a chemical moves through a protective clothing

material through diffusion, adsorption and desorption, and wicking.

TOP 08-2-503 20 February 2018

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APPENDIX B. ABBREVIATIONS.

ANOVA analysis of variance

AR Army Regulation

ATEC U.S. Army Test and Evaluation Command

BQL below quantifiable limit

CDD Capability Development Document

CoC chain of custody

CONOPS concept of operations

CPD Capability Production Document

CWA chemical warfare agent

DA Department of the Army

DEET N,N-diethyl-meta-toluamide

DI deionized

DoE design of experiment

DPG U.S. Army Dugway Proving Ground

DVB divinylbenzene

ECBC U.S. Army Edgewood Chemical Biological Center

EPA Environmental Protection Agency

FID flame ionization detector

FPD flame photometric detector

GC gas chromatography

HPLC high-performance liquid chromatograph

IAW in accordance with

APPENDIX B. ABBREVIATIONS

ICD Initial Capability Document

ISO International Organization for Standardization

LC liquid chromatograph

LVAP low volatility agent permeation

MQL method quantification limit

MS mass spectrometer

MSMS Tandem mass spectrometry

NIST National Institute of Standards and Technology

PAM pamphlet

PID photoionization detector

POLs petroleum, oil, and lubricants

PQL program quantification limit

PTFE polytetrafluoroethylene

QA quality assurance

QC quality control

RH relative humidity

RSD relative standard deviation

SEP System Evaluation Plan

SICN swatch identification control number

SOP Standing Operating Procedure

TEMP Test and Evaluation Master Plan

TIC toxic industrial chemical
TICN test item control number

APPENDIX B. ABBREVIATIONS.

TO test officer

TOP Test Operations Procedure

V&V verification and validation

VX persistent nerve agent

TOP 08-2-503 20 February 2018

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APPENDIX C. REFERENCES.

- 1. U.S. Army Edgewood Chemical Biological Center (ECBC), Edgewood, Maryland, *Development of a Contact Permeation Test Fixture and Method*, ECBC-TR-1141, April 2013.
- 2. U.S. Army Edgewood Chemical Biological Center (ECBC), Edgewood, Maryland, *Low-Volatility Agent Permeation (LVAP) Verification and Validation (V&V) Report*, ECBC-TR-1274, May 2015.
- 3. U.S. Army West Desert Test Center (WDTC), Dugway Proving Ground (DPG), Dugway, Utah, *Low-Volatility Agent Permeation (LVAP) Verification and Validation Report*, 9 June 2016.
- 4. TOP 08-2-501A, Permeation Testing of Materials With Chemical Agents or Simulants (Swatch Testing), 5 August 2013.
- 5. AR 385-10, The Army Safety Program, 27 August 2007 [Rapid Action Revision (RAR) 004, 4 October 2011].
- 6. DA PAM 385-61, Toxic Chemical Agent Safety Standards, 17 December 2008.
- 7. AR 50-6, Nuclear and Chemical Weapons and Materiel, Chemical Surety, 28 July 2008.
- 8. TOP 08-2-500A, Receipt Inspection of Chemical and Biological (CB) Materiel, 31 August 2017.

TOP 08-2-503 20 February 2018

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APPENDIX D. APPROVAL AUTHORITY.

Chemical, Biological, Radiological and Nuclear Defense Test and Evaluation Executive Approval



DEPARTMENT OF THE ARMY OFFICE OF THE DEPUTY UNDER SECRETARY OF THE ARMY 102 ARMY PENTAGON WASHINGTON, DC 20310-0102

DUSA-TE 22 January 2018

MEMORANDUM FOR SEE DISTRIBUTION

SUBJECT: Endorsement of Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) Test Operations Procedure (TTOP) 08-2-503 for Low Volatility Agent Permeation (LVAP) Swatch Testing

- Reference: Memorandum, DUSA-TE, 04 November 14, subject: Chemical and Biological Defense Program (CBDP) Test and Evaluation (T&E) Standards Development Plan.
- The Collective Protection (ColPro) and Individual Protection (IP) Capability Area Process Action Teams (CAPATs), now combined as the Warfighter Protection CAPAT, developed, coordinated and approved TTOP 08-2-503 in accordance with the reference.
- 3. I endorse this TTOP as a DoD T&E Standard for chemical defense in the areas of Collective Protection, Individual Protection, Decontamination, and Contamination Avoidance and encourage its broad use across all test phases. All T&E Standards are for government associated program access and use. You can access DoD T&E Standards via the following links: Army Knowledge Online (AKO), (https://www.us.army.mil/suite/files/22142943), and the TECMIPT site (http://www.amsaa.army.mil/TECMIPT/Standards.html).
- My point of contact for this action is Ms. Deborah Shuping, (703) 545-1119, deborah.f.shuping.civ@mail.mil.

for DAVID JIMENEZ
CBRN Defense T&E Executive

Encl

APPENDIX B. APPROVAL AUTHORITY.

CSTE-TM 20 February 2018

MEMORANDUM FOR

Commanders, All Test Centers
Technical Directors, All Test Centers
Directors, U.S. Army Evaluation Center
Commander, U.S. Army Operational Test Command

SUBJECT: Test Operations Procedure (TOP) 08-2-503 Low Volatility Agent Permeation (LVAP) Swatch Testing, Approved for Publication

 TOP 08-2-503 Low Volatility Agent Permeation (LVAP) Swatch Testing, has been reviewed by the U.S. Army Test and Evaluation Command (ATEC) Test Centers, the U.S. Army Operational Test Command, and the U.S. Army Evaluation Center. All comments received during the formal coordination period have been adjudicated by the preparing agency. The scope of the document is as follows:

This TOP provides the current standard methods for testing the permeation of low volatility chemicals, such as persistent nerve agent, through swatches of materials. Swatches can be taken from clothing or equipment that is new, with or without pretreatment(s), and that was previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure(s). These procedures are designed to be used for analyzing the material performance, manufacturing, and integration with other pieces of the protective ensemble.

- This document is approved for publication and will be posted to the Reference Library of the ATEC Vision Digital Library System (VDLS). The VDLS website can be accessed at https://vdls.atc.army.mil/.
- Comments, suggestions, or questions on this document should be addressed to U.S. Army Test and Evaluation Command (CSTE-TM), 6617 Aberdeen Boulevard-Third Floor, Aberdeen Proving Ground, MD 21005-5001; or e-mailed to usarmy.apg.atec.mbx.atec-standards@mail.mil.

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STEPHANIE J. HALCISAK Chief, Policy and Standardization Division

FOR

GARY R. GRAVES COL, FA Director, Test Management Directorate (G9) Forward comments, recommended changes, or any pertinent data, which may be of use in improving this publication to the Policy and Standardization Division (CSTE-TM), U.S. Army Test and Evaluation Command, 6617 Aberdeen Boulevard, Aberdeen Proving Ground, Maryland 21005-5001. Technical information may be obtained from the preparing activity: Director, West Desert Test Center, U.S. Army Dugway Proving Ground, ATTN: TEDT-DPW, Dugway, UT 84022-5000. Additional copies can be requested through the following website: http://www.atec.army.mil/publications/topsindex.aspx, or through the Defense Technical Information Center, 8725 John J. Kingman Rd., STE 0944, Fort Belvoir, VA 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.